ABSTRACT

In the dehydration step, a mixture comprising an organic amide solvent and a sulfur source is heated, vapor volatilized is guided to a distillation column, a fraction comprising the organic amide solvent as a principal component is refluxed into a reaction vessel, a fraction comprising water and hydrogen sulfide is cooled to discharge hydrogen sulfide that is not condensed by the cooling and reflux a part of water condensed into the distillation column, the remaining water is discharged, a relational expression between the total amount of water of an amount of water refluxed and an amount of water discharged without being refluxed, and an amount of hydrogen sulfide discharged from the reaction vessel is determined in advance, and an amount of hydrogen sulfide discharged from the reaction vessel is calculated out from a measured value of the total amount of water on the basis of the relational expression to control a charged molar ratio of the sulfur source to a dihalo-aromatic compound.